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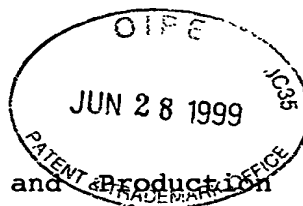
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**N. WILLIAMS****Certifying Officer**

[NAME OF DOCUMENT] Specification

[TITLE OF THE INVENTION]

Carbon Fiber Woven Fabric and Production Process  
Thereof



[SCOPE OF CLAIM FOR PATENT]

[Claim 1] A carbon fiber woven fabric having a volume resistivity of less than  $0.2 \Omega \cdot \text{cm}$ , a gas permeability of  $1,500 \text{ ml/cm}^2/\text{hr/mmHg}$  or more and a thickness of from 0.05 to 0.3 mm, wherein the carbon fiber oriented component in the thickness direction accounts for 1/3 (one third) or more of all carbon fibers.

[Claim 2] A process for producing the carbon fiber woven fabric described in claim 1, comprising calcining a woven fabric comprising a cellulosic fiber at 2,200 to 3,000°C in a non-oxidizing atmosphere.

[DETAILED DESCRIPTION OF THE INVENTION]

[0001]

[Technical Field to Which the Invention Belongs]

The present invention relates to a porous carbon sheet having a small thickness, which has excellent properties in the chemical resistance, electric conductivity, heat conductivity, gas permeability, handleability and the like and in which the in-plane gas permeability is uniform, more specifically, the present invention relates to a carbon fiber woven fabric useful as a porous carbon sheet for gas

diffusion of a solid polymer-type fuel battery.

[0002]

[Background Art]

In recent years, from the issue of environmental pollution by the exhaust gas of internal combustion engine of cars, an electric vehicle (EV) has been developed and for this, it is an essential matter to develop a fuel battery as a fuel source of EV. Thus, a higher performance and more compact fuel battery is being demanded.

The fuel battery includes various types of fuel batteries depending on the kind of the electrolytic solution used, such as alkali type, phosphoric acid type, carbonate type, solid electrolyte type and solid polymer type. Among these, the solid polymer-type fuel battery is being watched as a power source of EV and the like because it can work at a low temperature, is easy to handle and has a high output density.

As shown in Fig. 1, in the solid polymer-type fuel battery, an ion exchange membrane holding an appropriate water content and an electrically conducting porous diffusion sheet for allowing water or gas generated at the reaction of the battery to permeate are important. As the diffusion sheet, a sheet of porous carbon (hereinafter referred to as a "porous carbon sheet") is usually used.

The porous carbon sheet for gas diffusion used in the

solid polymer-type fuel battery is required to have properties such that the thickness is small, the gas permeability is high, the gas permeability within the sheet plane is uniform, the sheet is a porous sheet comprising open pores, the heat or electric conductivity is excellent, the volume resistivity in the thickness direction is low, and the handleability is good by having a strength large enough to facilitate the installing into an electrode cell.

[0003]

As the porous carbon sheet for gas diffusion used in a fuel battery heretofore known, JP-A-3-285873 (the term "JP-A" as used herein means an "unexamined published Japanese patent application") discloses a carbon sheet produced by impregnating a carbon fiber with a thermoplastic resin as a binder and calcination carbonizing it, which is used as a porous electrode sheet for a fuel battery, having a large number of pores in the direction intersecting the sheet plane or a porous carbon sheet for a fuel battery, improved in the air permeability, heat conductivity, compressive strength and the like.

Furthermore, as described, for example, in JP-B-2-58369 (the term "JP-B" as used herein means an "examined Japanese patent publication"), JP-B-2-23505 and JP-A-115970, the paper making process from organic fiber or cellulose is a most inexpensive production method for obtaining the

carbon sheet and this has been tested in some cases.

However, these techniques are disadvantageous in that the mass production is not available because impregnation with a thermoplastic resin and a calcination carbonization treatment at a high temperature are necessary.

Particularly, with the proceeding of development of electric vehicles or the like in recent years, the solid polymer-type fuel battery used therein is demanded to be more inexpensive and have higher performance and to this purpose, it is an essential matter that the porous carbon sheet for a gas diffusion layer is also more inexpensive and has higher performance. Furthermore, in order to realize reduction in the size or weight, a sheet having a thickness smaller than conventional sheets, in a fraction of mm or less is being demanded.

[0004]

[Problems to be Solved by the Invention]

The porous carbon sheet produced by the paper making process disclosed in the above-described publications has the following fundamental problems.

1) Since an organic fiber is milled into a sheet, the fibers are bent in the rolling process, that is, the fibers are aligned toward the direction parallel to the layer and therefore, the heat or electric conductivity in the layer-intersecting direction is low. Accordingly, an

electrically conducting carbon powder or the like must be blended at the milling to increase the electric conductivity but this causes problems, for example, reduces the mechanical strength or gas permeability.

2) Since the sheet formed passes through the step of impregnating it with a resin solution, the porous carbon sheet allows closed pores not in a small number to be present therein, as a result, a high gas permeability cannot be attained.

3) If a product having a small thickness is to be produced, the sheet milled as the original must be reduced in the thickness. This causes difficulties in the production by the paper making process and it becomes very difficult to maintain the in-plane uniformity of the sheet milled. As a result, the in-plane gas permeability becomes non-uniform.

4) The porous carbon sheet obtained by impregnating the sheet milled with a resin and heat-treating it, has little elasticity and is solid and fragile, therefore, the handleability in the working of assembling a battery is bad.

For solving these problems, conventional paper making processes are deficient and a new production process is demanded.

[0005]

[Means to Solve the Problems]

Under these circumstances, the present inventors have made extensive investigations and found that when a woven fabric comprising a cellulosic fiber is carbonization calcined as it is at from 2,200 to 3,000°C in a non-oxidizing atmosphere, a carbon fiber woven fabric having a thickness of from 0.05 to 0.3 mm, an electric resistivity of less than  $0.2 \Omega \cdot \text{cm}$  and a gas permeability of 1,500 ml/cm<sup>2</sup>/hr/mmAq or more can be obtained. In this carbon fiber woven fabric, the carbon fiber oriented component in the thickness direction accounts for 1/3 or more of all carbon fibers. Therefore, the fabric is suitably used as a porous carbon sheet for gas diffusion and the porous sheet using the fabric can be used in a fuel battery.

[0006]

[Mode for Carrying Out the Invention]

The present invention is described in detail below. The carbon fiber woven fabric is produced by carbonization calcining fibers comprising a cellulosic fiber. The original fiber yarn itself is soft and has a free directivity, therefore, the fibers are not bent as compared with a carbon fiber woven fabric obtained by weaving rigid carbon fibers. Furthermore, the orientation degree in the layer-intersecting direction is by far large as compared

with the fibers constituting the porous carbon sheet obtained by the above-described paper making process. In the present invention, the carbon fiber oriented component in the thickness direction accounts for 1/3 or more of all carbon fibers. With respect to the carbon fiber oriented component, the orientation system on the fine network plane and from the degree of the orientation, the orientation can be estimated by the observation through SEM. If the carbon fiber oriented component in the thickness direction is less than 1/3 of all carbon fibers, the heat conductivity and the electric conductivity in the layer-intersecting direction are deficient and the carbon sheet for gas diffusion used in the solid polymer-type fuel battery cannot have desired physical properties.

Thus, the carbon fiber woven fabric of the present invention is favored with excellent heat conductivity and electric conductivity in the layer-intersecting direction.

Furthermore, the carbon fiber woven fabric of the present invention is produced without passing through the step of immersing it in a resin solution, accordingly, pores of the fiber itself are not closed and the open pore ratio increases. At the same time, the heat melting or curing is dispensed with and the starting material is a cellulosic fiber which does not melt but carbonizes in the solid phase, therefore, the fibers are prevented from



adhering to each other and need not be compressed by a roll or the like and the fiber orientation mode of the green fabric before the calcination is reflected as it is in the carbon fiber woven fabric as a product. In addition, the final sheet can have excellent strength and good handleability.

Also, the carbon fiber woven fabric of the present invention is not impregnated with resin, carbon powder or the like as required in the paper making process, therefore, is suitable for producing a sheet having a small thickness.

The carbon fiber woven fabric of the present invention has a low carbonization ratio and accordingly, a great shrinkage occurs during the carbonization calcining. Therefore, a cellulosic fiber having a thickness larger than the thickness required must be used as a starting material and the shrinkage ratio is necessary to be examined in advance by a test.

The starting material for use in the present invention is a woven fabric, therefore, the number of pores in the plane and the in-plane uniformity necessary for the gas permeation can be controlled by the weaving method. Considering the large distribution in the in-plane density of a paper obtained by the paper making process, since the uniformity of commercially available fabrics is reflected on the gas permeability in the plane after the calcination,

the woven fabric of the present invention is naturally favored with excellent uniformity.

The woven fabric comprising a cellulosic fiber as a starting material according to the present invention may be a cloth of natural cellulose fiber such as cotton and hemp, or a cloth of artificial cellulose fiber such as viscose silk and acetate silk.

The weaving may be performed by various weaving methods such as plain weaving and twill weaving and among these, in view of the in-plane uniformity, plain weaving is preferred because warp and weft yarns are the same in the size. However, the woven fabric is not particularly limited and commercially available woven fabrics can be freely used.

In the present invention, the cellulosic fiber woven fabric is calcined at from 2,200 to 3,000°C. The lower the calcination temperature, the higher the permeability. However, if the calcination temperature is less than 2,200°C, the carbonization insufficiently proceeds and a sufficiently low resistivity cannot be obtained, whereas if it exceeds 3,000°C, the starting material fiber burns.

The calcination must be performed in a non-oxidizing atmosphere such as argon gas and nitrogen gas.

[0017]

[Examples]

The present invention is described in greater detail below by referring to the Examples.

A commercially available plain weave cotton cloth having a thickness of 0.3 mm (yarn size: 0.2 mm) was used as a green sample for the test.

The green sample was cut into a 400 mm square, interposed between graphite plates, and carbonization calcined in an electric furnace at 900°C over 1 week in an argon gas atmosphere.

The carbonized sheet had a thickness of 0.20 mm, a size of 330 mm square and good appearance, where the shape of the plain weave cotton cloth as the green sample remained.

This carbonized sheet was interposed between graphite plates and graphitized by burning it at a temperature shown in Table 1 over 1 day in an argon gas atmosphere.

The graphitized sheet had a thickness of from 0.15 to 0.16 mm, a size of from 320 to 322 mm square and good appearance, where the shape of the green sample similarly remained. Each of these carbon fiber woven fabrics having a size of about 320 mm square was equally divided into 25 pieces and various physical properties were measured.

The pore size and porosity were measured by a mercury

press fitting method and the volume resistivity is measured by a dc four probe method and shows a value measured in the direction parallel to the layer. The gas permeability was calculated from the differential pressure obtained when an air was permeated through a 50-cm<sup>2</sup> sheet at a rate of 3,000 ml/min, according to the following formula:

gas permeability =

$$3,000 \text{ (ml/min)} / 60 \text{ (min)} / 50 \text{ (cm}^2\text{)} / \text{ differential pressure (mmAq)}$$

The measured values of various physical properties of 25 samples are shown in Table 2.

As a reference, the values in the case of sheet produced by a conventional paper making process disclosed in JP-B-58369 are also shown in Table 2.

The compressive strength of the sheets obtained in Examples was from 40 to 50 Kgf/cm<sup>2</sup> and this was large enough for the use as a carbon sheet.

[0008]

[Table 1]

Temperature Condition in Graphitization

	Graphitization °C
Example 1	2300
Example 2	2200
Example 3	2400
Example 4	2600
Comparative Example 1	1000

[Table 2]

Various Measured Values (the values shown by a range indicate maximum and minimum of 25 samples)

	Bulk Density g/cm <sup>3</sup>	Pore Size μm	Porosity %	Volume Resistivity Ω·cm	Gas Permeability ml/cm <sup>2</sup> /hr/ mmAq
Example 1	0.32-0.31	65-60	79-76	0.12-0.11	1760-1630
2	0.30-0.30	62-59	77-75	0.11-0.11	1660-1630
3	0.29-0.28	60-58	75-74	0.10-0.10	1650-1600
4	0.28-0.28	59-58	74-73	0.09-0.09	1620-1580
Comparative Example 1	0.30-0.31	37-35	55	0.17-0.16	3600-3400
Reference Value					
1	0.27	36	69	0.14	-
2	0.27	58	59	0.16	-
3	0.29	62	59	0.17	-

[0009]

[Effects of the Invention]

According to the present invention, a carbon fiber woven fabric having excellent performance as a porous carbon sheet for gas diffusion used in a fuel battery can be obtained using only an inexpensive commercially available cellulose fiber woven fabric as a starting material and only by a step of calcination without passing through a complicated step such as paper making, impregnation with resin and calcining.

[BRIEF DESCRIPTION OF DRAWING]

[Fig. 1]

Fig. 1 is a cross-sectional view showing the fundamental structure of a solid polymer-type fuel battery using the porous sheet of the present invention.

[Description of Numerical References]

- 1 separator sheet with grooves
- 2 porous anode gas diffusion sheet
- 3 anode catalyst layer
- 4 ion exchange membrane
- 5 cathode catalyst layer
- 6 porous cathode diffusion sheet

[NAME OF THE DOCUMENT] Abstract

[SUMMARY]

[PROBLEM TO BE SOLVED]

To produce a carbon fiber woven fabric having a small thickness for use as a porous carbon sheet for gas diffusion or the like of a solid polymer-type fuel battery, using an inexpensive starting material through a simple process.

[MEANS TO SOLVE THE PROBLEM]

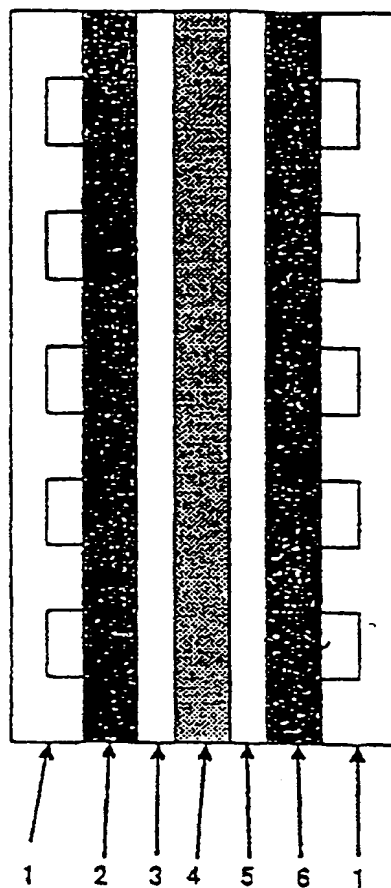
By calcining a cellulosic fiber woven fabric at from 2,200 to 3,000°C in a non-oxidizing atmosphere, a carbon fiber woven fabric having a volume resistivity of less than 0.2  $\Omega \cdot \text{cm}$ , a gas permeability of 1,500 ml/cm<sup>2</sup>/hr/mmAq or more and a thickness of from 0.05 to 0.3 mm can be obtained, wherein the carbon fiber oriented component in the thickness direction accounts for 1/3 (one third) or more of all carbon fibers.

[SELECTED DRAWING] None.

[NAME OF DOCUMENT]

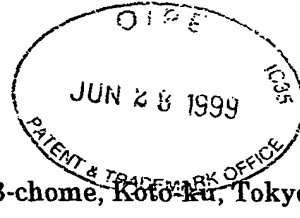
Drawing

[Fig. 1]





## DECLARATION



I, Atsuko Ikeda, residing at 26-2-906, Ojima 3-chome, Koto-ku, Tokyo, Japan, do hereby certify that I am conversant with the English and Japanese languages and am a competent translator thereof. I further certify that to the best of my knowledge and belief the attached English translation is a true and correct translation made by me of U.S. Provisional Patent Application No. 60/128,054 filed on April 7, 1999.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 2nd day of June, 1999

  
Atsuko Ikeda

# PROVISIONAL APPLICATION COVER SHEET

Attn: Assistant Commissioner for Patents, Washington, DC 20231

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53(c).

Docket Number	P53878	Type a plus sign (+) inside this box -->	+
<b>INVENTOR(s)/APPLICANT(s)</b>			
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<b>TITLE OF THE INVENTION (280 characters max)</b>			
CARBON FIBER WOVEN FABRIC AND PRODUCTION PROCESS THEREOF			
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<b>ENCLOSED APPLICATION PARTS (check all that apply)</b>			
<input checked="" type="checkbox"/> Specification* *Japanese Language <input checked="" type="checkbox"/> Drawing(s)		Number of Pages <u>9</u> Number of Claims <u>2</u> Number of Sheets <u>1</u>	
		<input type="checkbox"/> Small Entity Statement <input type="checkbox"/> Other (specify)	
<b>METHOD OF PAYMENT (check one)</b>			
<input type="checkbox"/> A check or money order is enclosed to cover the Provisional Application filing fees. The Office is also directed and authorized to charge or credit any difference or overpayment to Deposit Account No. 19-4880.			Provisional Application Filing Fee Amount \$150.00
<input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge filing fees and/or credit any difference or overpayment to Deposit Account No. 19-4880.			
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government. <input checked="" type="checkbox"/> No. <input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are:			
Respectfully submitted, 		Date: April 7, 1999	
Waddell A. Biggart <small>Type or Printed Name</small>		Registration No. 24,861	

☐ Additional Inventors are being named on separately numbered sheets attached hereto.

**PROVISIONAL APPLICATION FILING ONLY**

【書類名】 明細書

【発明の名称】 炭素繊維織布およびその製造方法

【特許請求の範囲】

【請求項1】 体積固有抵抗 $0.2$ オーム・cm未満、ガス透過率 $1500$ cc/cm<sup>2</sup>/hr/mmAq 以上の特性を持つ厚さ $0.05 \sim 0.3$ mm、かつ炭素繊維の厚さ方向の配向成分が炭素繊維全体の $1/3$ 以上あることを特徴とする炭素繊維織布。

【請求項2】 セルロース質繊維からなる織布を非酸化性雰囲気下、 $2200^{\circ}\text{C} \sim 3000^{\circ}\text{C}$ にて焼成することを特徴とする請求項1記載の炭素繊維織布の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】

本発明は、耐薬品性、電気伝導性、熱伝導性、ガス透過性、ハンドリング性等に優れ、かつ面内のガス透過性が均一である肉薄の多孔質カーボンシートに関し、特に固体高分子型燃料電池のガス拡散用多孔質カーボンシートに有用なる炭素繊維織布に関する。

【0002】

【従来の技術】

近年の自動車等内燃機関の排ガスによる環境汚染問題から、電気自動車（EV）が開発されているが、このためには、その燃料源として燃料電池の開発が必須であり、より高性能、小型の燃料電池が求められている。

燃料電池には、使用する電解液の種類によりアルカリ型、リン酸型、熔融炭酸塩型、固体電解質型、固体高分子型等、種々のタイプの燃料電池があるが、低温で稼働でき、扱い易く、かつ出力密度の高い固体高分子型がEV等の動力源として注目を集めている。

固体高分子型燃料電池は、図1に示すように、適度な水分を保持するイオン交換膜とともに、それらの電池反応時に発生する水、ガスを通過処理するための導電性の多孔質拡散シートが重要となる。この拡散シートは、通常多孔質なカー

ボンの薄板が用いられる。(以下多孔質カーボンシートと呼ぶ。)

該固体高分子型燃料電池に用いられるこれらガス拡散用多孔質カーボンシートに要求される特性としては、肉薄であること。ガス透過性が良く、かつシート面内のガス透過性が均一であること。開気孔からなる多孔質シートであること。熱および電気伝導性に優れており、かつ厚さ方向の体積固有抵抗が低いこと。更には、電極セルに組み込むために適度の強度を持ちハンドリング性にすぐれていること等が求められている。

#### 【0003】

今までに、燃料電池用ガス拡散用多孔質カーボンシートとして、特開平3-285873、特開平5-254957等にて炭素繊維にバインダーとして熱硬化性樹脂を含浸し、焼成、炭化して製造したカーボンシートにおいて、シート面の垂直方向に多数の微細孔を持つ燃料電池用多孔質電極板、空気透過率、熱伝導率、圧縮強度等を改善した燃料電池用多孔質カーボンシートが開示されている。

また例えば特公平2-58369、特公平2-23505、特公平7-115970に示されるように有機繊維、セルロースをもとに、抄紙法による製造が最も安価に該カーボンシートを得る製造法である旨が開示され一部でもテストされてきた。

しかしこれらは、熱硬化性樹脂の含浸、高温炭化焼成処理が必要であるため、量産性に欠けコストが高いという問題があった。

特に、近年になり電気自動車等の開発の進展に伴い、これらに用いられる固体高分子型燃料電池の更なる、安価、高性能化が求められ、同時にガス拡散層用多孔質カーボンシートも、より安価、かつ高性能化が必須となっている。また小型化、軽小化を進めるため0.数mm以下の従来より薄いものが求められている。

#### 【0004】

##### 【発明が解決しようとする課題】

上記に開示されているような抄紙法による多孔質カーボンシートに関しては、以下のような根本的な問題を抱えている。

- 1) 有機繊維を抄紙してシートにするため、抄紙ロール工程で繊維が寝てし

まう。つまり繊維方向が沿層方向に並んでしまうため、貫層方向の熱、および電気伝導性が劣る。この為、導電性のカーボン粉末等を抄紙時に配合し電気伝導度を上げてやる必要があり、この結果、機械的強度やガス透過性を低減させる等の不具合があった。

- 2) 抄紙したシートを樹脂液に浸す工程を経るため、少なからず多孔質カーボンシート中に、有効に作用しない閉気孔を内在させてしまう。このため、ガス透過性が向上しない。
- 3) 肉薄の製品を作ろうとすると、もととなる抄紙シートを薄くせねばならず、抄紙法としては製造が難しくなる。これは抄紙シートの面内均一性を保つのが非常に困難となり、この結果として面内のガス透過性の不均一を生じることとなる。
- 4) 抄紙シートを樹脂に浸し、熱処理した多孔質カーボンシートは、弾性が少なく固く脆いため、電池の組み立て作業時のハンドリング性が良くない。

これらの要求応えるには、従来の抄紙法では問題があることから新たな製法改良が求められていた。

#### 【0005】

##### 【課題を解決するための手段】

本発明者らは、前記状況に鑑み鋭意検討した結果、セルロース質繊維からなる織布をそのまま、非酸化性雰囲気下2200℃以上、3000℃以下に炭化焼成することにより、厚さ0.05～0.3mm、電気比抵抗0.2オーム・cm未満、ガス透過率1500cc/cm<sup>2</sup>/hr/mmHg以上の炭素繊維織布が得られ、またこの炭素繊維の厚さ方向の配向成分が炭素繊維全体の1/3以上を占め、ガス拡散用多孔質カーボンシートの用途に好適であり、これを燃料電池に使用することができることを、見いだした。

#### 【0006】

##### 【発明の実施の形態】

さらに詳細に本発明について説明すれば、本発明による炭素質繊維織布は、セルロース質繊維からなる繊維を炭化焼成しているため、元の繊維の糸自体が柔

らかく自由な方向性を持っておるため、剛直な炭素繊維を織った炭素繊維織布に比べても繊維は寝ておらず、また前記の抄紙法による多孔質カーボンシートを構成する繊維に比べても遥かに貫層方向の配向度合いが大きい。本発明において炭素繊維の厚さ方向の配向成分は、炭素繊維全体の $1/3$ 以上である。炭素繊維の配向成分は、微細組織の網面の配向方式とその配向度合いから配向度をSEM観察等により推定することができる。炭素繊維の厚さ方向の配向成分が炭素繊維の全体の $1/3$ 以下では、貫層方向の熱伝導性、電気伝導性が不足し、固体高分子型燃料電池のガス拡散用カーボンシートに所望の物性が得られない。

従って、本発明による炭素繊維織布は貫層方向の熱伝導性、電気伝導性に優れる。

また、本発明による炭素繊維織布は、樹脂液に浸す工程が無いため、繊維自体の気孔を閉鎖することなく、開気孔率が向上する。同時に、樹脂の熱溶融、硬化が無く、原料は溶融せず固相炭化するセルロース質繊維を出発点としているため、繊維同士が固着せず、ロール等での圧縮を必要としないため、焼成前のグリーン織布の有している繊維配向形態をそのまま製品である炭素繊維織布中に反映すると同時に、最終的に強度に優れ、ハンドリング性が良い。

また、抄紙法のような樹脂、カーボン粉末等の含浸等が無いため、肉薄のシートを製造するのに適している。

本発明の炭素繊維織布は炭化率が低いこともあり、炭化焼成中の収縮が大きい。従って所要とする厚さより厚めのセルロース質繊維を原料とすることが必要で、収縮率を事前のテストにて調べておく必要がある。

本発明に使用する原料は、織り布であるため、ガス透過のための面内の孔数、面内の均一性は織り方によりコントロール可能であり、抄紙法によるペーパーの面内の密度のばらつきの大きさを考えると、市販の織布の均一性が焼成後の面内のガス透過性に反映されるので、当然本発明は、その均一性に優れている。

なお、本発明の原料のセルロース質繊維からなる織布は、綿、麻等の天然セルロース繊維の布でも、ビスコース人絹、アセテート人絹等の人造セルロース繊維の布でもかまわない。

織り方は、平織り、アヤ織り等各種考えられるが、面内の均一性の点からは

縦横糸太さが同一の平織りが好ましいが、何ら制限されるものではなく、これらは容易に市販の織布が利用できる。

本発明においては、セルロース質繊維織布を2200℃～3000℃にて焼成する。焼成温度は、低いほうが透過率は大きくなるが、焼成温度が2200℃未満では、炭化が不十分であり十分に低い固有抵抗値が得られず、3000℃を超えると原料の繊維が燃えてしまう。

なお、焼成はアルゴンガス、窒素ガス等の非酸化性雰囲気で行う必要がある。

【0007】

#### 【実施例】

以下、実施例により本発明を更に詳細に説明する。

厚さ0.3mmの市販平織り綿布（糸太さ0.2mm）を用いてテスト用グリーンサンプルとした。

グリーンサンプルを400mm角に裁断し、黒鉛板に挟持した状態で電気炉にてアルゴン雰囲気下1週間をかけて900℃に炭化焼成した。

該炭化シートは厚さ0.20mm、330mm角の外観良好なグリーンサンプルである平織り綿布の形態を残していた。

この炭化シートを黒鉛板に挟持し、表1に示す各温度にてアルゴン雰囲気下1日かけて焼き上げ黒鉛化した。

黒鉛化されたシートは同様に厚さ0.15～0.16mm、320～322mm角の外観良好なグリーンサンプルと同様の形態を残していた。この約320mm角の各炭素繊維織布を均等に25分割し諸物性を測定した。

孔径、気孔率については、水銀圧入法により、体積固有抵抗は直流4端子法による沿層方向の測定値である。ガス透過率については、3000ml/minの空気を該シート50cm<sup>2</sup>に透過させたときの差圧から以下の式により計算した。

$$\text{ガス透過率} = 3000 \text{ (ml/min)} / 60 \text{ (min)} / 50 \text{ (cm}^2\text{)} / \text{差圧 (mmHg)}$$

表2に該25個のサンプルの諸物性の測定値を示す。

参考値として従来の抄紙法により製造した特公平2-58369に開示された値を表2に併記する。

また実施例で得られたシートの圧縮強度は、40～50 Kg f / cm<sup>2</sup>でカーボンシートとして充分使用に耐えるものであった。

【0008】

【表1】

黒鉛化温度条件

	黒鉛化温度   ℃
実施例1	2300
実施例2	2200
実施例3	2400
実施例4	2600
比較例1	1000

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【表 2】

各種測定値（範囲で示したものは25個の最大、最小値）

	嵩密度 g/cm <sup>3</sup>	孔径 μm	気孔率 %	体積固有抵抗 オーム・cm	ガス透過率 cc/cm <sup>2</sup> / hr/mmAq
実施例	0.32	65	79	0.12	1760
1	~0.31	~60	~76	~0.11	~1630
2	0.30	62	77	0.11	1660
	~0.30	~59	~75	~0.11	~1630
3	0.29	60	75	0.10	1650
	~0.28	~58	~74	~0.10	~1600
4	0.28	59	74	0.09	1620
	~0.28	~58	~73	~0.09	~1580
比較例	0.30	37		0.17	3600
1	~0.31	~35	55	~0.16	~3400
参考値					
1	0.27	36	69	0.14	—
2	0.27	58	59	0.16	—
3	0.29	62	59	0.17	—

【0009】

## 【発明の効果】

本発明によれば、安価な市販のセルロース繊維織布のみを原料として、抄紙、樹脂含浸、焼成等の複雑な工程を経ず焼成のみの工程で、燃料電池用のガス拡散用多孔質カーボンシートとして優れた性能を有する炭素繊維布が得られる。

## 【図面の簡単な説明】

## 【図 1】

本発明の多孔質シートを使用する固体高分子型燃料電池の基本構成を示す断面図。

【符号の説明】

- 1 溝付きセパレーター板
- 2 多孔性アノードガス拡散シート
- 3 アノード触媒層
- 4 イオン交換膜
- 5 カソード触媒層
- 6 多孔質カソード拡散シート

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【書類名】 要約書

【要約】

【課題】 固体高分子型燃料電池のガス拡散用多孔質カーボンシート等に使  
用される肉薄の炭素繊維織布を安価な原料、簡単な工程にて製造する。

【解決手段】 セルローズ質繊維織布を非酸化性雰囲気にて2200～3000  
℃にて焼成することにより、体積固有抵抗0.2オーム・cm未満、ガス透過率  
1500cc/cm<sup>2</sup>/hr/mmHg以上の特性を持ち、厚さ方向の配向成分  
が炭素繊維全体の1/3以上ある厚さ0.05～0.3mmの炭素繊維織布が得  
られる。

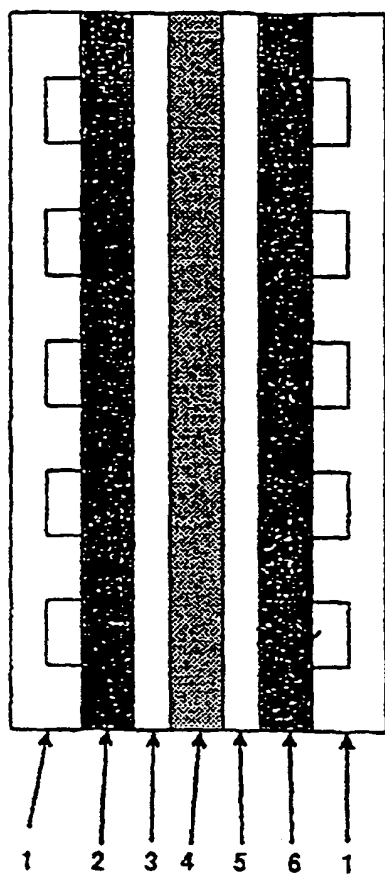
【選択図】 なし

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【書類名】

図面

【図 1】



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